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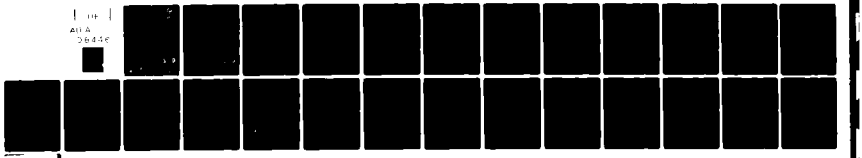

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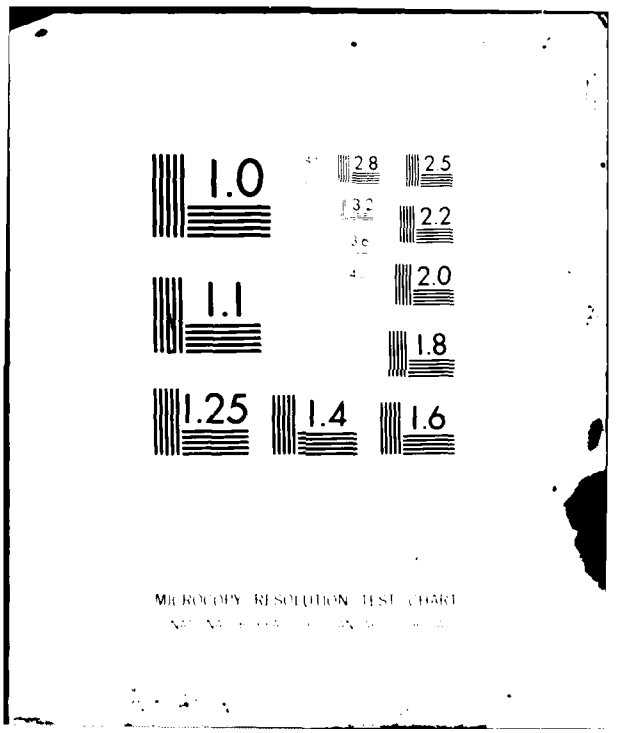
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CRACK VELOCITY DEPENDENCE OF ELECTRON EMISSION DURING FRACTURE OF FILLED ELASTOMERS

by

J. T. Dickinson and L. C. Jensen

Physics Department

Washington State University

Pullman, WA 99164

November 1981

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

We examine the electron emission accompanying the fracture of filled elastomers, which in previous work we have shown to be intense and due predominately to interfacial failure. In this study we show that there is a strong velocity dependence on the rate of electron emission during the propagation of a crack in filled silicon rubber and polybutadiene filled with glass beads. We propose that the concentration of free radicals produced by crack propagation increases rapidly with crack velocity which in turn produces more intense electron emission by a chemi-emission mechanism.

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ABSTRACT

In this paper we examine the electron emission accompanying the fracture of filled elastomers, where in previous work we have shown the emission to be intense and due predominately to interfacial failure. In this study we show that there is a strong velocity dependence on the rate of electron emission during the propagation of a crack in filled silicon rubber and polybutadiene filled with glass beads. This dependence cannot be explained by a simple proportionality between emission rate and rate of surface area production. We propose that the concentration of free radicals produced by crack propagation increases rapidly with crack velocity which in turn produces more intense electron emission by a chemi-emission mechanism.

Introduction

Fracto-emission (FE) is the emission of particles (electrons, ions, neutral species, and photons) during and following crack propagation. Previous studies[1-9] have shown that a wide range of materials produce FE when fractured including fiber reinforced composites, filled and unfilled elastomers, and the peeling of pressure-sensitive adhesives. Systems in which adhesive or interfacial failure occur were found to emit with very high intensity and long-lasting emission after rupture. This enhanced, long-lasting emission was attributed to the types of chemical species produced by interfacial failure (as compared to cohesive failure) and the high degree of electrical charging that occurs with interfacial failure.[7-9]

As yet unpublished results recently obtained on a number of filled elastomer systems shows that the decay we observe in the electron emission (EE) and positive ion emission (PIE) after fracture follows the kinetics of free radical decay similar to that observed by ESR methods in a number of polymers. We therefore suggest that the observed EE (and PIE) is strongly linked to the production and subsequent decay of such species. It appears that for systems involving adhesive failure, a high density of radicals are produced at a free surface, the latter being essential for the ejection of charge into the vacuum. Thus as the rate of production of surface increases, one would naturally expect the rate of EE to increase. The major question we wish to answer here is: how does the rate of EE relate to V_c, the crack velocity?

Experimental

The details of our experimental procedures are given in Ref. 7. Samples of filled polybutadiene (BR) were provided by The University of Akron Institute of Polymer Science. The consisted of Diene 35 NFA, Firestone Tire

and Rubber Co. mixed with dicumyl peroxide at a concentration of 0.05 percent by weight and with glass beads, 30-95 μ in diameter, at a concentration of 34% by volume. Samples of a commercial 50 Durometer Red Silicon Rubber (SI) were also studied. This material was highly filled (about 50% by volume) with mineral particles of irregular shape, typically 1 to 10 μ in diameter, and with very fine Fe_2O_3 powder of a particle size too small to measure under a SEM. SEM photos of fractured surfaces for both these systems showed a high concentration of exposed particles/beads free of elastomer indicating that interfacial failure was extensive.

The typical BR sample was 2 mm x 4.5 mm and the SI samples were 1.5 mm x 15 mm. These were supported in the pulling mechanism with clamps with initial separation of 6 mm. The top edge of the samples was notched in the center and loaded in tension so that the crack propagated in the vicinity of the detector. Experiments were carried out in a vacuum chamber at a pressure of 2-4 x 10^{-6} Torr. The residual gases consisted primarily of CO, H_2O, and CO_2. Some samples were tested in an ion pumped vacuum system at a pressure of 10^{-8} Torr to determine the influence of the background gases; no detectable differences were observed.

The detector was a Galileo Electro-Optics Model 4830 channel electron multiplier (CEM) positioned 6 cm from the sample. This particular CEM has relatively low gain, but is capable of higher count rates. This prevented saturation of the detector during times of high emission rates. The front of the CEM was biased at +300 V for efficient collection of electrons. The pulse output (10 ns pulse width) of the CEM was amplified and fed to a 100 mHz discriminator whose output was counted as a function of time by a multichannel scalar; the typical dwell time per channel was 1 ms, and the duration of the crack propagation changed from a few ms to several seconds.

Simultaneous to the EE measurements, a standard video system viewed the sample and recorded the crack position vs. time. Measurements from the crack position on each frame (1/60 s) allowed calculation of V_c. This method is capable of measuring $V_c \leq 20$ cm/sec, corresponding to slow-medium crack growth.

Results

The general features of the observed EE accompanying and following fracture can be seen on a slow time scale as shown in Fig. 1. The noise level is typically less than 2 counts per second. The EE at the peak occurs during crack propagation; i.e., the maximum rate of emission occurs when the crack is moving. Following rupture, the EE decays in a complicated fashion. This part of the decay curve is fit nicely by models involving the creation and decay of free radicals similar to results of ESR studies on free radicals produced by fracture and γ-radiation.[10-13]

Figure 2 shows the EE on a faster time scale focusing on the events during crack propagation. The strain rate for these events was approximately 1.0/s. The arrow represents the instant the crack transversed the entire sample. In both filled BR and SI the emission started relatively weak and grew to a maximum at the instant of separation.

The video tape of the fracture event revealed in 1/60s time intervals the motion of the crack. In both materials, initial application of strain produced very slow crack growth and the formation of a U-shaped crack-tip. The tip gradually accelerated with rapid acceleration occurring during the final moments of crack propagation. This rapid acceleration was more pronounced in the filled BR samples. Figure 3 presents the measured V_c vs. time, corresponding to the EE curves of Fig. 2, for both materials; the lines shown are smooth curves drawn through the data.

Matching corresponding data, we obtain the resultant EE intensity vs. V_c curves shown in Fig. 4 and 5. Figure 4 represents the first part of the data, prior to separation, on a linear scale. The data for SI are from two samples showing that the trend is reproducible. The data in Fig. 5 are from the same experiments plotted on a log scale and extending to the video frame just prior to separation. The last instant of rupture occurred too rapidly (within one frame) to measure a velocity.

Both sets of curves show that the V_c dependence of EE is very strong with a possible transition to exponential behavior at higher velocities. Both materials have similar shape, differing primarily in intensity. The curve has positive, non-zero slope (monotonically increasing) at all velocities, and approaches zero emission much like a linear function ($EE \propto V_c$). If the EE intensity were simply proportional to the rate of surface area produced for all V_c, then the EE count rate would remain linear with V_c. Since the dependence is much stronger, it indicates that the excitation mechanisms leading to EE are dependent on V_c.

We can measure the EE during fracture on much smaller time scales and thus explore faster fracture events even though currently we cannot make corresponding V_c measurements. For a strain rate approximately 180 times faster we show the resulting EE vs. time curves (on a log scale) for the same materials in Fig. 6. The arrows show when final separation occurred--a few channels of after-emission are also shown. One sees that the emission is still rising and in the case of BR took a dramatic jump, presumably due to a very high V_c at the end of crack propagation. Obviously, V_c measurements at these higher rates are of considerable interest.

The EE response to a series of periodic increments in strain is shown in Fig. 7 for a SI sample. The increments in strain were approximately 0.03%.

Each increment led to acceleration followed by deacceleration of the crack which in turn led to peaks superimposed on a smooth rise in EE intensity.

As a confirming test to establish that EE depends on V_c we fractured filled BR with constant detector geometry and efficiency at two strain rates: 1.0/s and 180/s. Comparing peak emission heights, total emission before and/or after separation, we find that the faster strain rate led to approximately a factor of 10 more emission, due to the higher V_c.

Discussion

Before modeling the shape of the EE vs. V_c curves we feel it necessary to make more measurements to confirm their basic features. However, at this point we would like to offer a few ideas relating these results to known phenomena associated with fracture of elastomers. First, such fracture is known to produce free radicals in such materials.[10-14] Second, a filled elastomer frequently exhibits detachment from filler particles in the highly strained region near the crack tip.[15] Our earlier work[7-9] has shown that such interfacial failure produces enhanced EE, presumably due to higher concentrations of free radicals and charge. SEM photographs show that in both filled materials, SI and BR, the degree of interfacial failure is very high. We therefore suggest that the resulting velocity dependence of EE is because the rate of failure at the surface of the filler particles determines the concentration of free radicals on the surfaces of the interface; i.e., the more rapid the detachment the higher the concentration of free radicals.

This higher concentration may be a consequence of the fact that more rapid loading does not allow local molecular stresses in the region of the interface to be relieved via visco-elastic relaxation mechanisms which at room temperature typically require milliseconds. Thus more rapid loading causes more primary bond scission which in turn would lead to a higher rate of

free-radical formation. Such results have been observed in a number of homogeneous polymers[16] using ESR to detect the free radicals.

Summary

We have examined the EE during crack propagation in filled elastomers and found that there is a strong dependence of EE on V_c. This dependence is not due to simply a higher rate of creating surface area which would lead to a linear relation between EE and V_c. The rapid growth in EE intensity with V_c is due to the production of higher concentrations of excitations, presumably free radicals, produced from the scission of primary bonds in the region of the interface between the filler particles and the elastomer. Clearly more work is required to further quantify the results, extend them to higher V_c, and relate our measurements to the microscopic events occurring in the fracture of polymers.

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We wish to thank Dr. A. N. Gent, University of Akron Institute of Polymer Science for supplying the polybutadiene samples and for very helpful discussions. We also thank M. K. Park for his assistance in the laboratory.

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FIGURE CAPTIONS

Fig. 1. Electron emission from the fracture of Red Silicone Rubber (SI) and Polybutadiene (BR) filled with 30-95 um glass beads. Crack motion was occuring during the rise and the peak of the emission.

Fig. 2. Electron emission during fracture of SI and BR with glass beads. The arrow indicates when fracture was completed, i.e., when separation occurred.

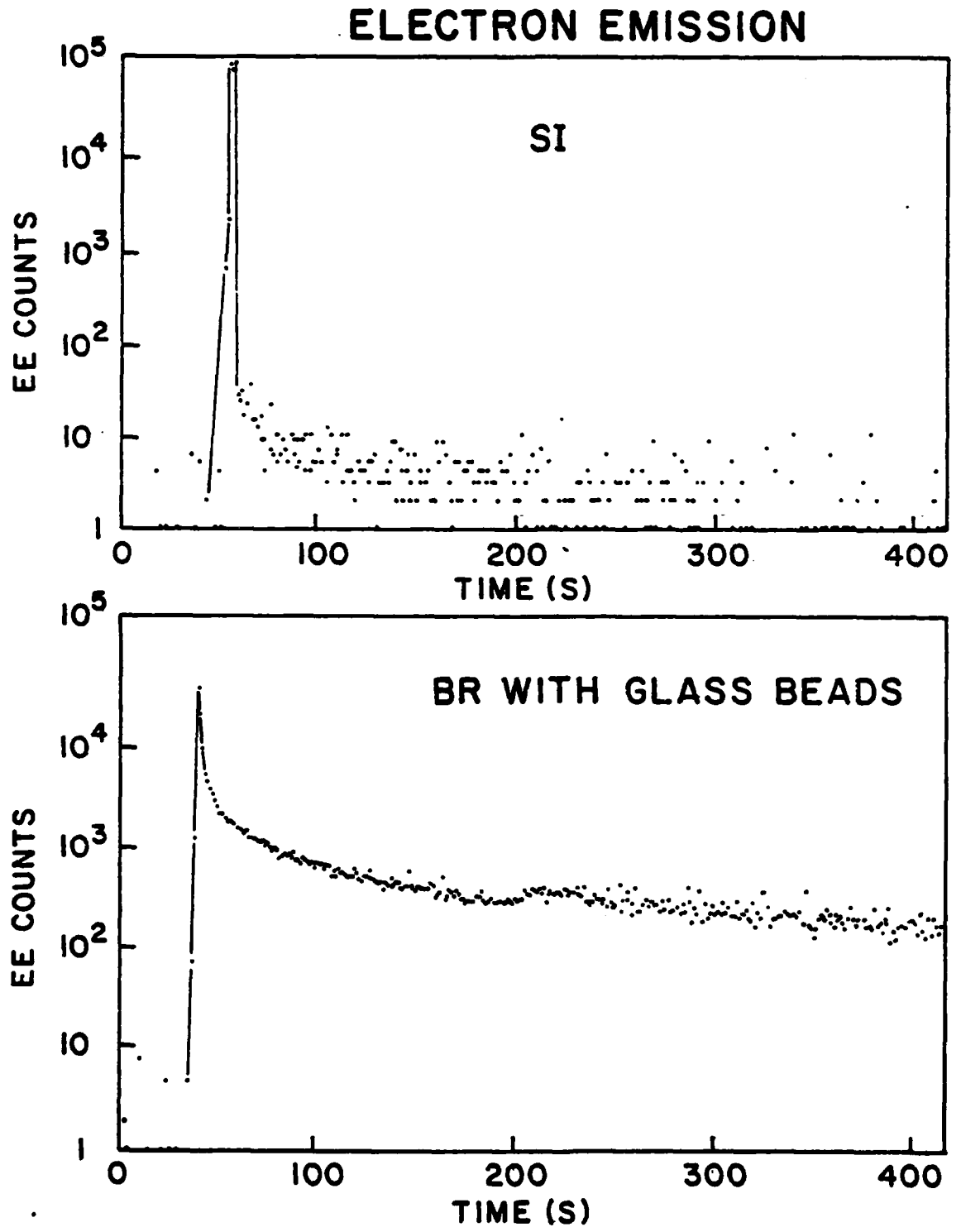
Fig. 3. Measured values of the crack velocities vs time corresponding to the emission curves in Fig. 2.. The lines are smooth curves drawn through the data points.

Fig. 4. Electron emission vs crack velocity on a linear scale for the first part of the velocity scale. The data shown for SI are from two samples.

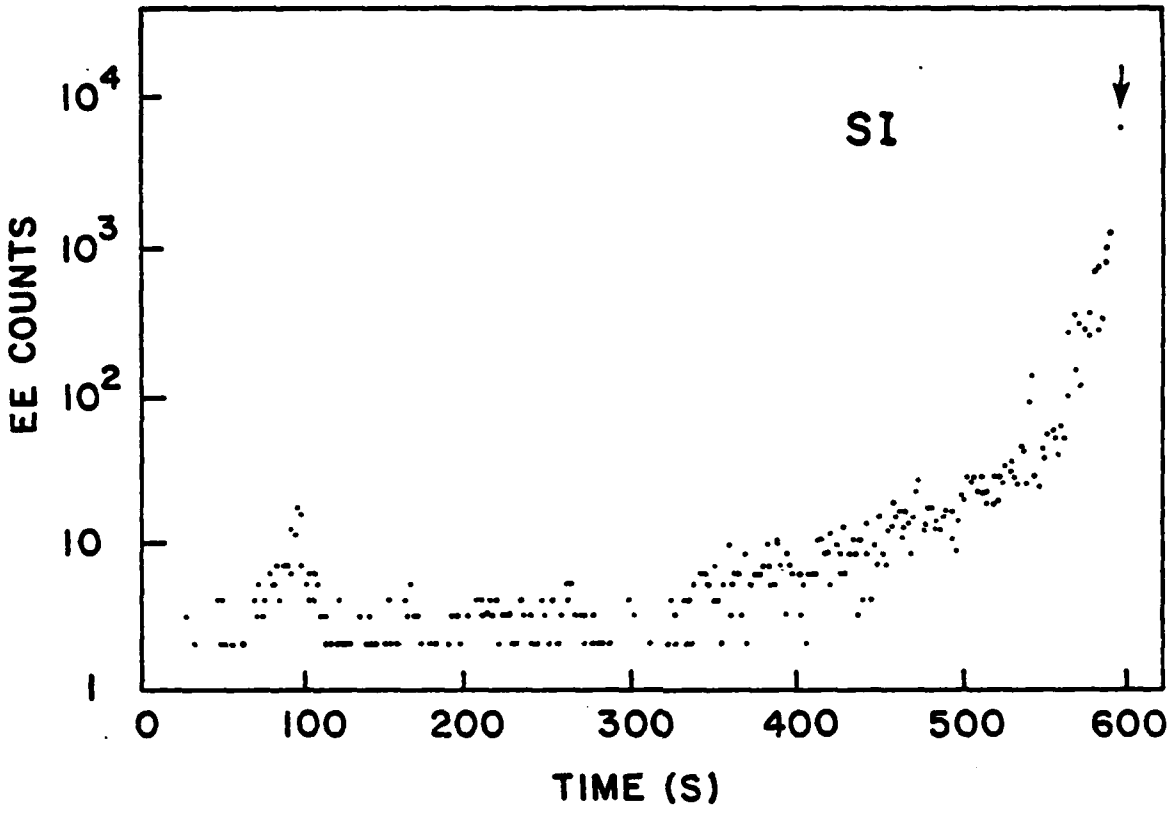
Fig. 5. Electron emission vs crack velocity on a log scale extended to higher velocities. The last data points at higher velocities are just prior to separation.

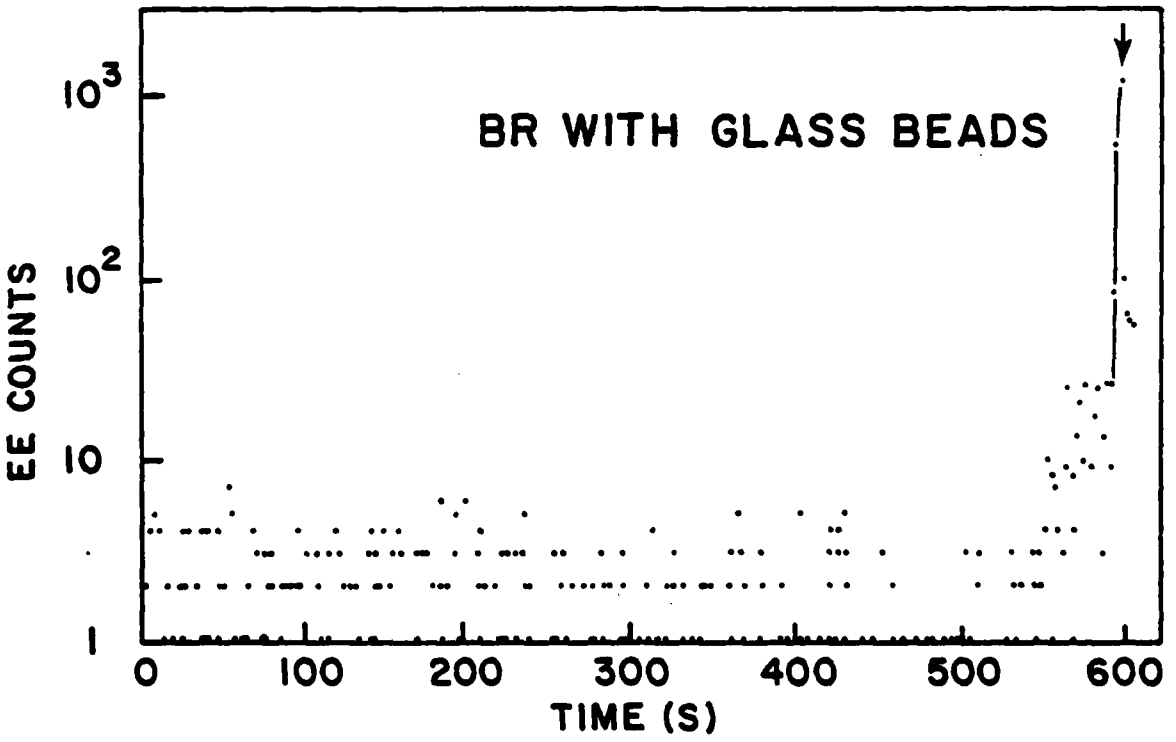
Fig. 6. Electron emission vs time for SI and BR filled with glass beads for samples strained at higher rates. We could not measure V_c for these fracture events using the video system.

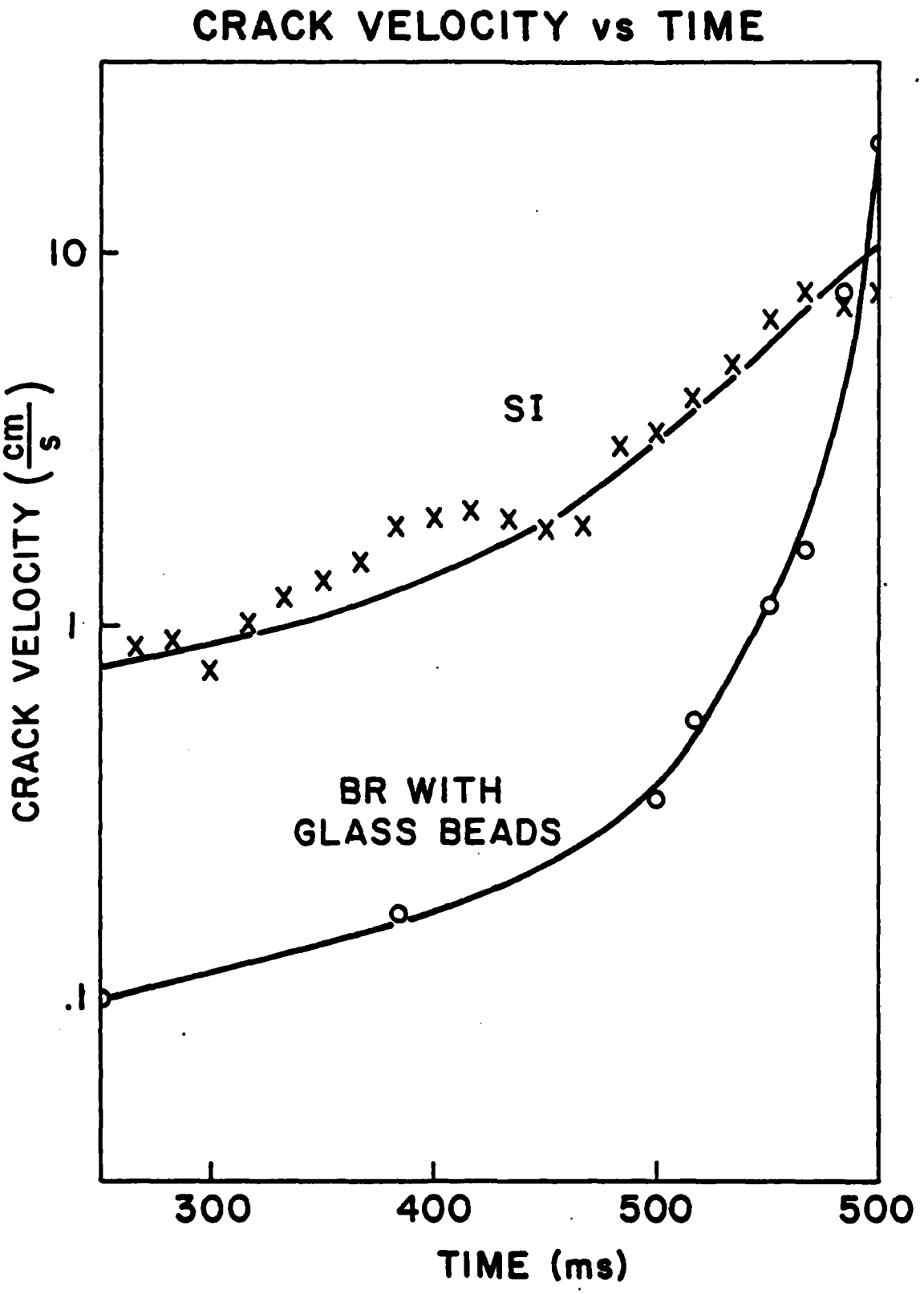
Fig. 7. Electron emission during crack propagation in SI where the strain was incremented periodically on a slow time scale. Each rise in emission corresponded to an increment in strain.

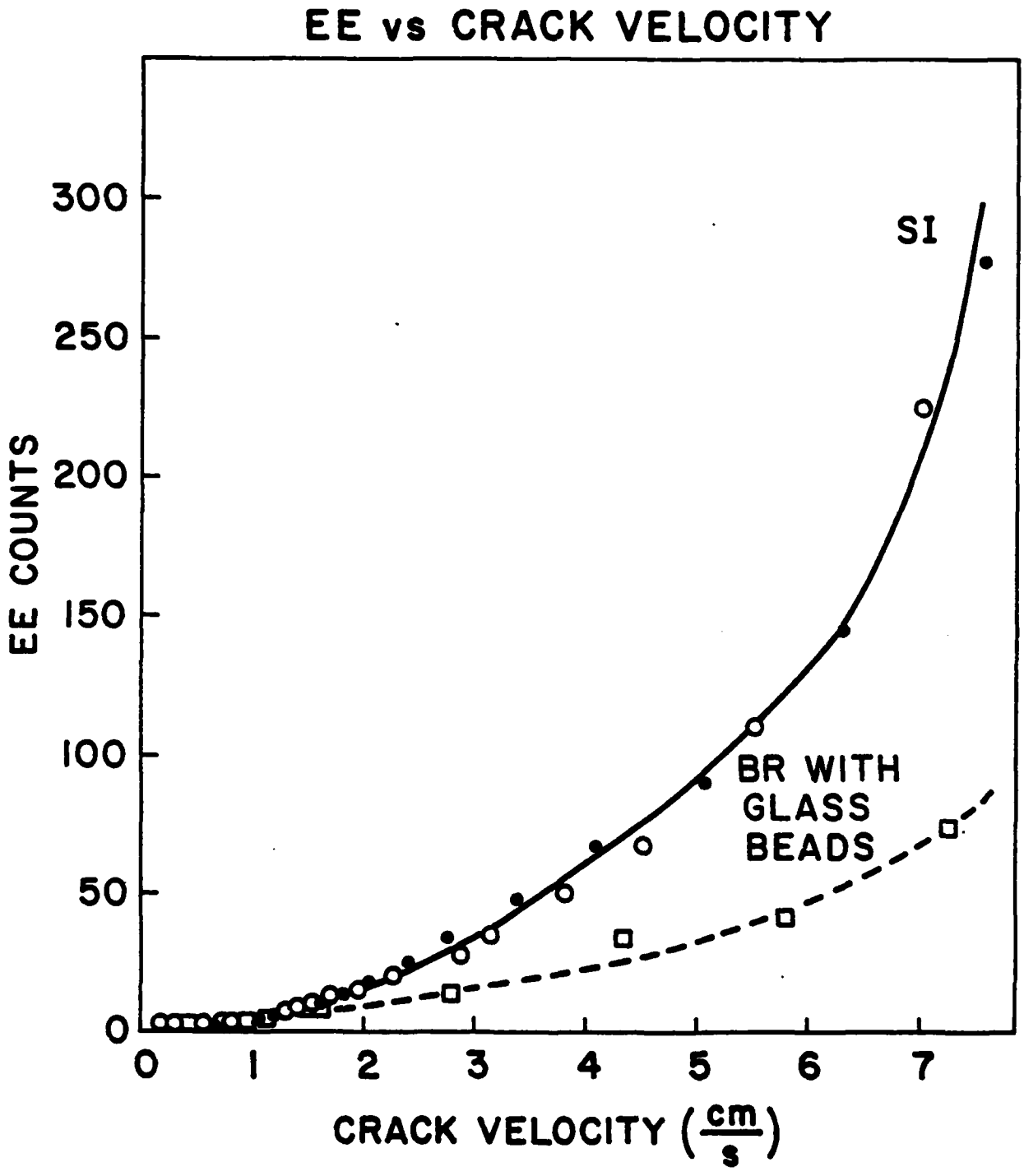

ELECTRON EMISSION
SI
EE COUNTS
10^5
10^4
10^3
10^2
10
1
0
100
200
300
400
TIME (S)
BR WITH GLASS BEADS
EE COUNTS
10^5
10^4
10^3
10^2
10
1
0
100
200
300
400
TIME (S)

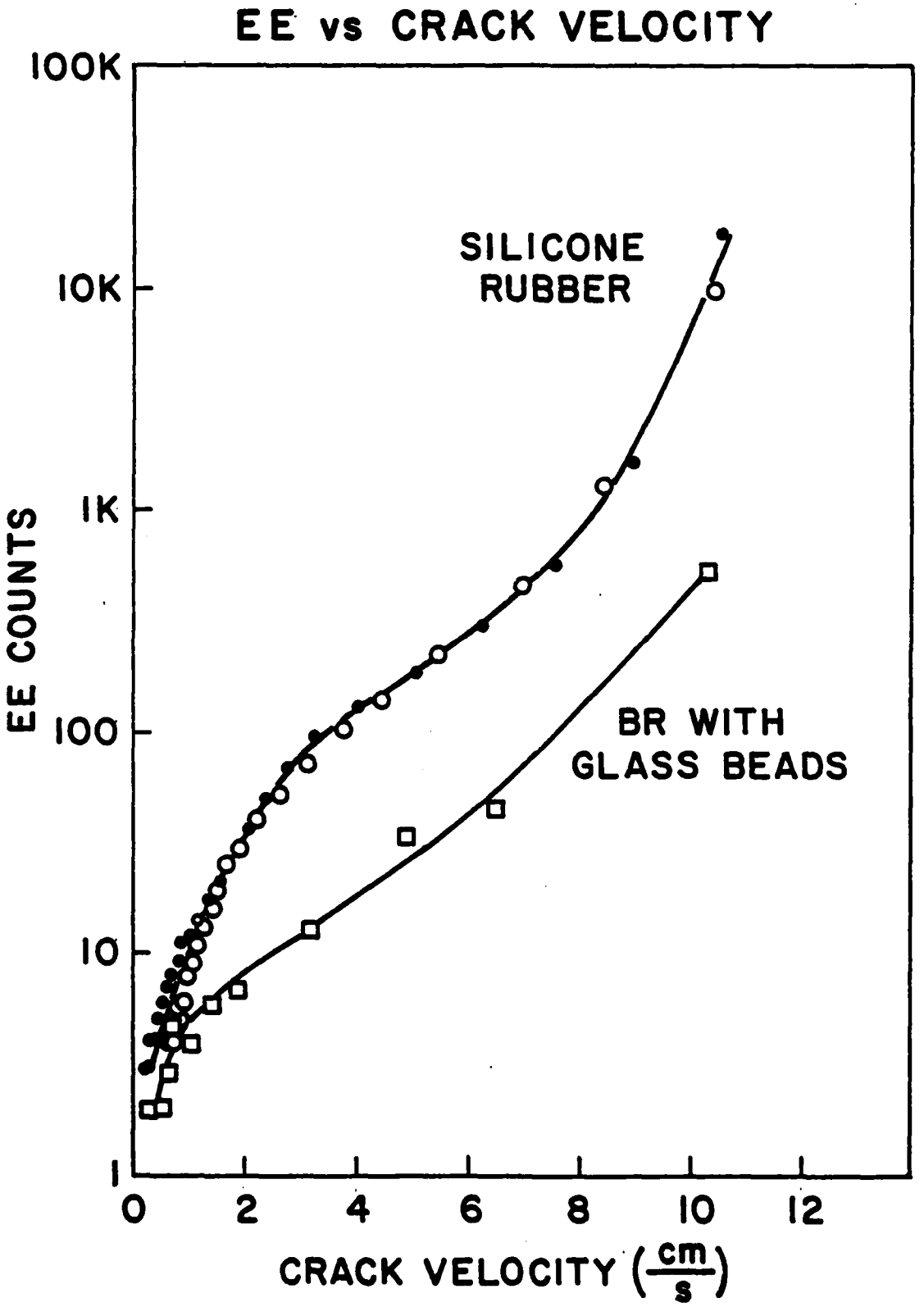
EE DURING FRACTURE

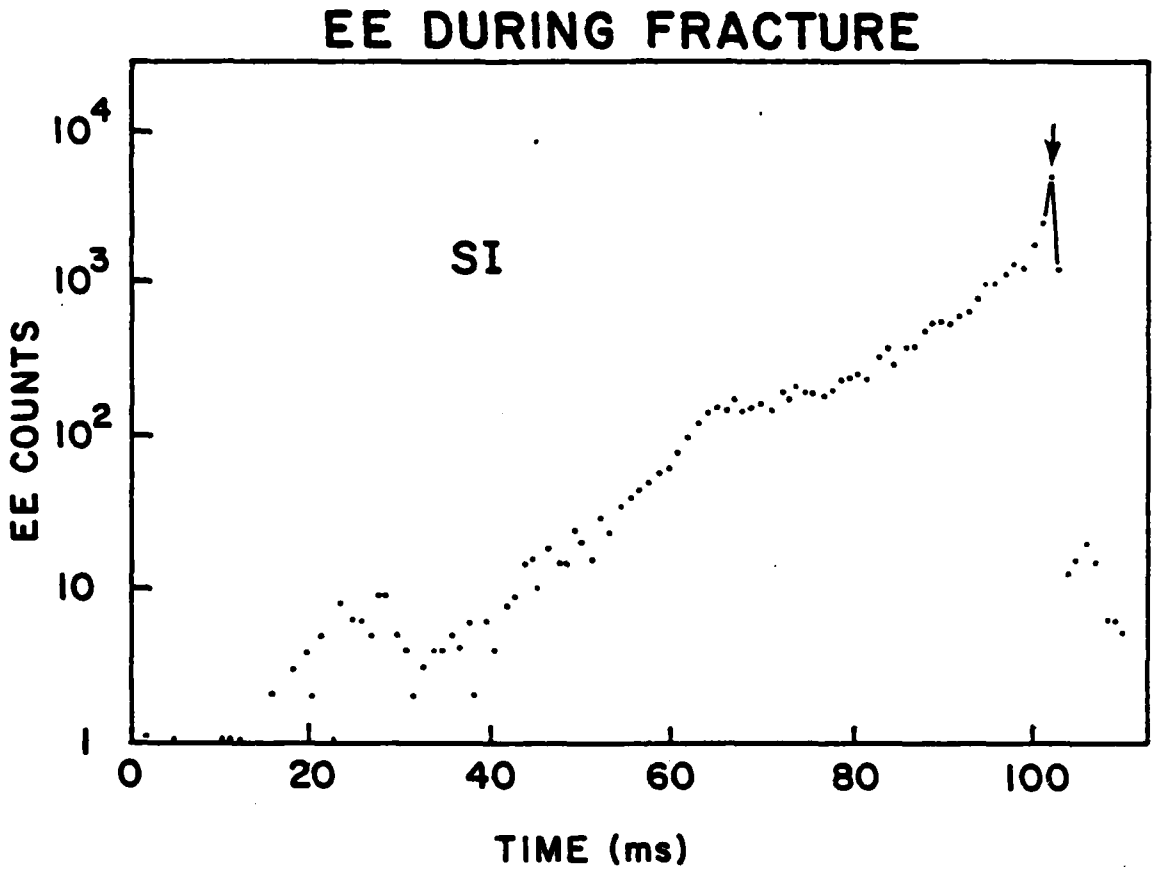


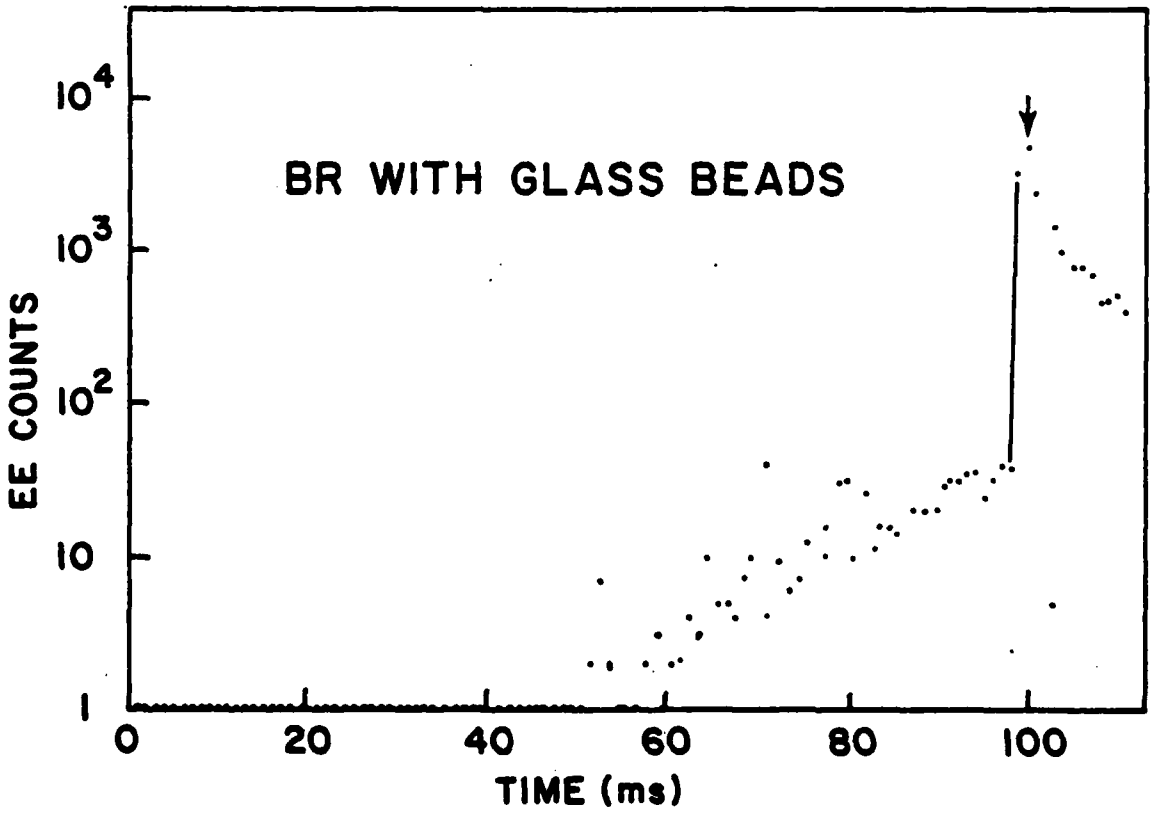


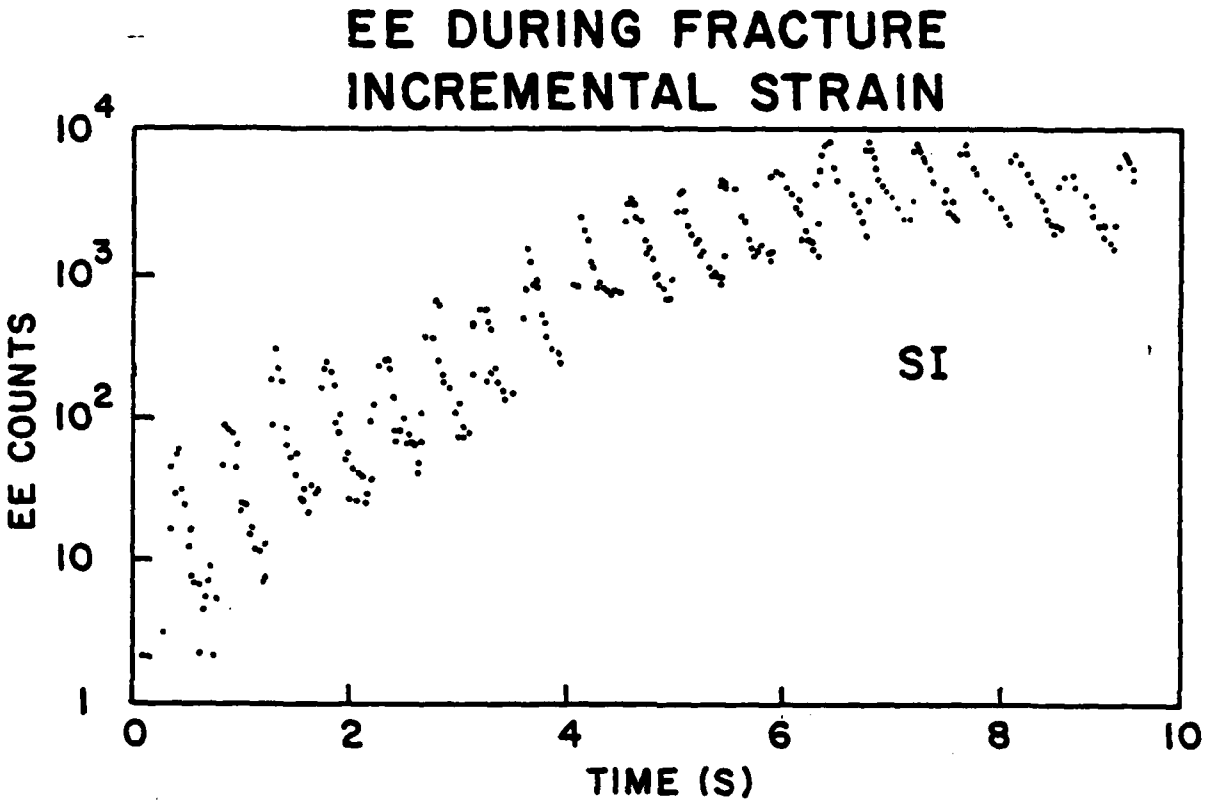

CRACK VELOCITY vs TIME
10
1
.1
CRACK VELOCITY (cm/s)
SI
BR WITH
GLASS BEADS
300
400
500
500
TIME (ms)


EE vs CRACK VELOCITY
EE COUNTS
300
250
200
150
100
50
0
0
1
2
3
4
5
6
7
CRACK VELOCITY (cm/s)
SI
BR WITH
GLASS
BEADS


EE vs CRACK VELOCITY
100K
10K
1K
100
10
1
EE COUNTS
SILICONE
RUBBER
BR WITH
GLASS BEADS
0
2
4
6
8
10
12
CRACK VELOCITY (cm/s)


EE DURING FRACTURE
SI
EE COUNTS
10^4
10^3
10^2
10
1
0
20
40
60
80
100
TIME (ms)


BR WITH GLASS BEADS
EE COUNTS
10^4
10^3
10^2
10
1
0
20
40
60
80
100
TIME (ms)


EE DURING FRACTURE
INCREMENTAL STRAIN
EE COUNTS
10^4
10^3
10^2
10
1
SI
0
2
4
6
8
10
TIME (S)

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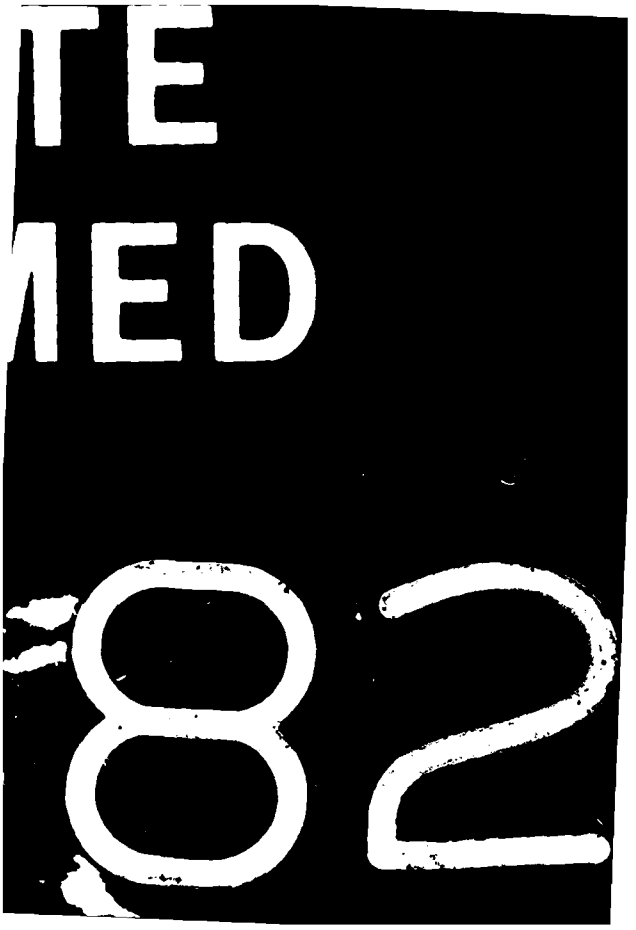
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